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Influence of Mineral Oil and Additives on Microhardness and Surface Chemistry of Magnesium Oxide {001} Surface

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Summary

The surface of magnesium oxide was examined by X-ray photoelectron spectroscopy. Also examined were the hardness and the dislocation widths around the Vickers indentations on the magnesium oxide surface. Surface analysis and static indentation experiments were conducted with the cleaved magnesium oxide {001} surface in various environments. The magnesium oxide bulk crystals were cleaved into the appropriate specimen size along the {001} surface in laboratory air, in nitrogen gas, or in degassed mineral oil with and without an additive. Indentations were made on the cleaved surface in the desired environment without exposing the specimen surface to any other environment. The various additives examined contained sulfur, phosphorus, chlorine, or oleic acid. Indentations were made in the magnesium oxide with a diamond indenter. All indentation experiments were conducted with a load of 0.1 N at room temperature. The time in contact was 20 sec.

The hardness and dislocation widths around an indentation on the magnesium oxide cleaved in mineral oil without and with phosphorus or oleic acid additives were almost the same as those of the surfaces cleaved in air or nitrogen gas. The sulfur-containing additive exhibited the highest hardness and smallest dislocation widths evidencing plastic deformation; the chlorine-containing additive exhibited the lowest hardness and largest dislocation widths evidencing plastic deformation.

Hydrocarbon and chloride (MgCl_2) films were formed on the magnesium oxide surface as a result of a chlorine-containing additive interacting with the cleaved surface. A chloride film was responsible for the lowest measured hardness.

Introduction

The presence of small amounts of impurities in metals and nonmetals and/or the presence of adsorbed surface-active ions or molecules on their surfaces can alter the surface activity of these materials (refs. 1 to 5).

Tribological properties such as adhesion, friction, resistance to deformation, wear, and lubrication of solid surfaces in contact are extremely dependent on the impurities in the bulk materials and on the environment. The microhardness of solids would be affected by such impurities and environments because microhardness is

one of the measurements used to indicate the extent of plastic deformation. The effect of the environment on microhardness has been studied by many investigators with a variety of nonmetals such as CaF_2 , Al_2O_3 , and LiF (refs. 5 to 8). Although considerable effort has been put forth in determining the environmental effects on the hardness of nonmetals, neither the fundamentals of the surface chemistry involved nor the tribological properties of the solid surfaces in a variety of environments are clearly understood.

This report examines the surface chemistry and microhardness of single-crystal magnesium oxide in various environments. The magnesium oxide bulk crystals were cleaved to a specimen size along the {001} surface in laboratory air, in nitrogen gas, or in degassed mineral oil with and without an additive. Indentations were made on the cleaved surface in the desired environment without exposing the specimen surface to any other environment. Various additives contained sulfur, phosphorus, chlorine, or oleic acid.

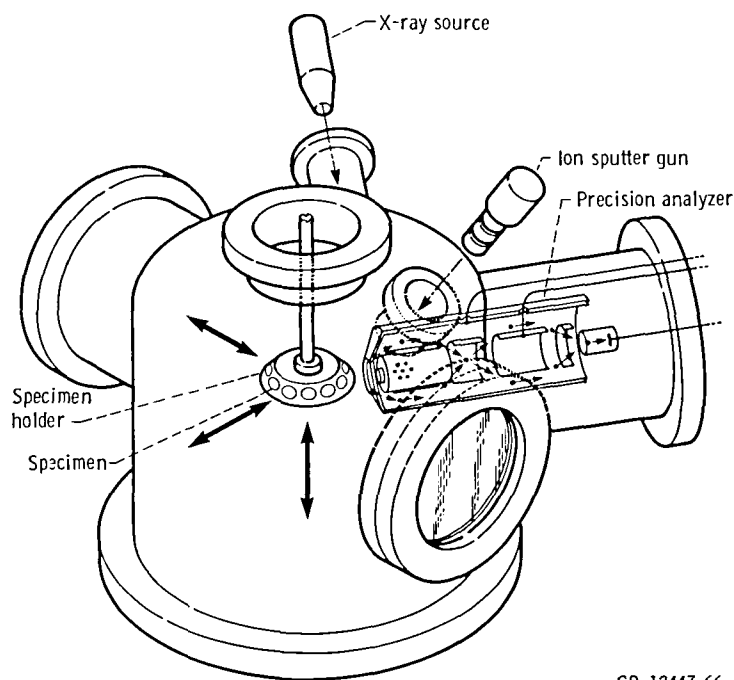
The surface chemistry of magnesium oxide crystals was analyzed by X-ray photoelectron spectroscopy (XPS). XPS provides analysis of surface composition. Microhardness (Vickers) indentation experiments were conducted on magnesium oxide {001} surfaces with a diamond indenter. All indentation experiments were conducted with a load of 0.1 N at room temperature. The time in contact was 20 sec.

Materials

The arc-melted, single-crystal magnesium oxide used in this investigation was 99.99 percent pure. Magnesium oxide is an ionic crystal with a rock-salt structure. This structure consists of two interpenetrating face-centered cubic lattices, one made up of positive ions and the other of negative ions (ref. 9). Because magnesium oxide is highly ionic, slip does not occur on the close-packed {001} cube planes, but instead on the {011} cube diagonals (ref. 9).

Apparatus

Two apparatuses were used in this investigation. One was an XPS system contained in an ultra-high-vacuum chamber; it is shown schematically in figure 1. The figure indicates the major components, including the electron energy analyzer, the X-ray source, and the ion gun used



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Figure 1. - Schematic representation of the X-ray photoelectron spectrometer.

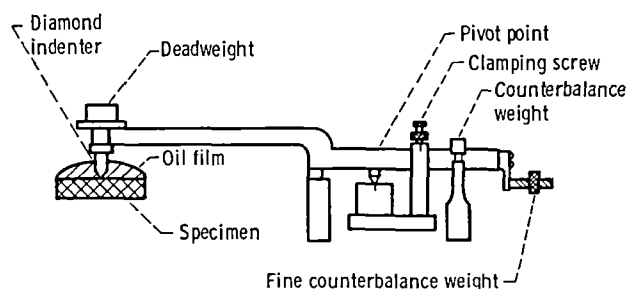


Figure 2. - Microhardness apparatus.

for ion sputter etching. The X-ray source contained a magnesium anode. The specimens were mounted on the end of the specimen probe at an angle of 60° from the analyzer axis. The X-ray source was located at an angle of 79° from the analyzer axis. The ion gun was located at an angle of 72° to the analyzer axis.

The second apparatus was a microhardness (Vickers) tester capable of measuring indentation hardness in nitrogen or other controlled environments (fig. 2). Although the indenting could be done automatically, the vibration generated by the electric motor and gears in the tester introduced remarkable error in the indentation. To avoid this error, indenting was done manually with the tester fixed on a vibration-free table. A diamond indenter was attached to one end of a precision-balanced rod. The load was applied by placing deadweights on a pan on top of this end of the rod. The specimen holder was moved vertically by hand until the specimen was in contact with the diamond indenter. The entire apparatus was housed

in a plastic box. The atmosphere in the box could be closely controlled.

Experimental Procedure

Specimen Preparation and Indentation Experiments

The magnesium oxide bulk crystals were cleaved to the appropriate specimen size along the {001} surface in laboratory air or nitrogen gas at room temperature in the plastic box that housed the microhardness tester by using a knife edge and hammer. The bulk crystals were also immersed in degassed mineral oil (in vacuum) with and without an additive at room temperature and cleaved to the specimen size along the {001} surface, again by using a knife edge and hammer. The specimens were mounted on the microhardness tester as shown in figure 2 without exposing them to any other environment. The indentation diagonals were parallel to the {001} directions. The mineral oil with an additive contained 2 wt % additive. Table I presents the formulas for the additives used in this investigation and their common chemical names.

X-Ray Photoelectron Spectroscopy

The cleaved specimens were placed in the vacuum chamber (fig. 1) without cleaning, and the system was evacuated and subsequently baked out to obtain a pressure of 10 nPa (10^{-10} torr). The surfaces were examined with the XPS at a pressure of 10 nPa.

TABLE I. - CHEMICAL FORMULAS
OF ADDITIVES

Surface active species	Formula	Common chemical name
Phosphorus	$(C_7H_7O)_3PO$	Tricresyl phosphate
Sulfur	$[CH_3(CH_2)_3]_2S$	Dibutyl sulfide
Chlorine	$CH_3(CH_2)_{17}Cl$	Octadecyl chloride
Carbonyl group	$C_{18}H_{34}O_2$	Oleic acid

To clean specimens by argon ion sputtering, the ion gun was outgassed for 2 min at a degas emission current of 20 mA. The sublimation pump was then flashed on for about 2 min at 48 A. The ion pumps were then shut off. The inert gas, argon, was bled in through the leak valve to the desired pressure of 7 mPa with the ion pump turned off. The ion sputtering was performed with a beam energy equal to 3000 eV at 20-mA emission current with the argon pressure maintained at 7 mPa for the desired sputtering time. The ion beam was continuously rastered over the specimen surface. After sputtering, the system was evacuated to a pressure of 30 nPa or lower, and then the surface was examined with XPS.

To obtain reproducible results, a strict standardization of the order and time of recording was used. The instrument was regularly calibrated. The analyzer calibration was determined by assuming the binding energy for the gold $4f_{7/2}$ peak to be 83.8 eV; that is, the gold $4f_{7/2}$ level was used as the reference line. All survey spectra (scans of 1000 or 1100 eV) were taken at a pass energy of 50 or 100 eV. The instrumentation resolution was thus 1 eV at room temperature. The magnesium $K\alpha$ X-rays were taken with an X-ray power source of 400 W

(10 kV, 40 mA). The narrow scans of the Mg_{2p} , O_{1s} , Cl_{2p} , and S_{2p} , which were just wide enough to encompass the peaks of interest, were obtained with a pass energy of 25 eV at room temperature.

During analysis, insulating specimens tend to acquire a steady-state charge of as much as several volts. To evaluate the charging effect and to determine accurately the energy of the peaks, the following approach was taken in this investigation: The position of the C_{1s} line was measured. This line is caused by the hydrocarbons introduced from the laboratory environment (ref. 10) and thus nearly always present on the specimens. The C_{1s} line on unsputtered inert metals such as gold appears at 284.6 eV; so any shift from this value can be taken as a measure of the static surface charge. Thus the value of 284.6 eV for the C_{1s} line was used as the reference line for static surface charging on the magnesium oxide.

To determine the relative concentrations of the various constituents on the magnesium oxide surface, the XPS measurement was qualified by using peak area sensitivity factors. A simplified expression to determine the atomic concentration C_x of any element x is given in the following equation (ref. 10):

$$C_x = \frac{I_x/S_x}{\sum_i I_i/S_i} \quad (1)$$

where I_x is the relative peak area of photoelectrons from element x . The atomic sensitivity factors S_x were calculated according to theoretical photoelectron cross sections, the kinetic energy dependence of the electron energy analyzer, and an average value for the dependence of the electron escape depth on kinetic energy (ref. 10). The relative peak areas of photoelectrons were arrived at by integrating the area under the peaks.

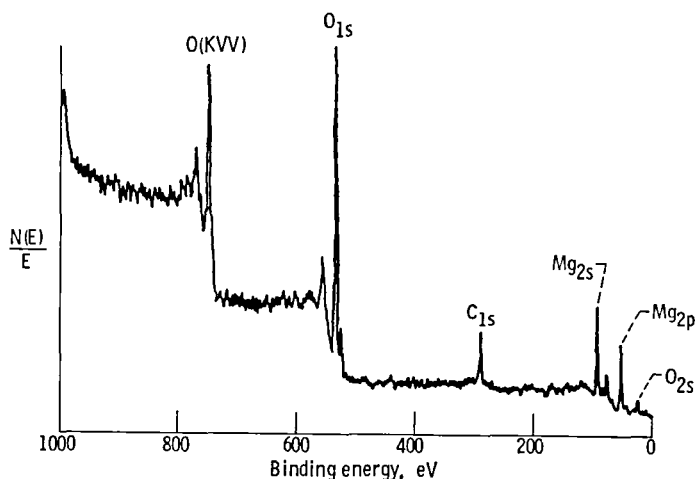


Figure 3. - Survey spectra of magnesium oxide {001} surface cleaved in laboratory air.

Results and Discussion

Surface Chemistry and Hardness of Magnesium Oxide

Survey spectra (1000-eV scans) were taken of a magnesium oxide surface cleaved in laboratory air. The spectrum taken before argon ion sputter cleaning indicated a carbon contaminant on the magnesium oxide surface, as shown in figure 3. The spectrum taken after argon ion sputter cleaning reveals no carbon on the magnesium oxide surface.

The XPS spectra of the Mg_{2p} and O_{1s} obtained from narrow scans on the magnesium oxide {001} surfaces are shown in figure 4. The Mg_{2p} and O_{1s} photoelectron lines for the as-cleaved crystal after bakeout and before argon ion sputter cleaning peaked at 50.2 and 531.0 eV, respectively. The O_{1s} photoelectron lines have two peaks. The higher peak indicates the presence of magnesium oxide itself; the lower peak is believed to be due to an oxide formed from the interaction of oxygen adsorbed on the magnesium oxide crystal. After ion sputter cleaning, however, the lower peak (from the foreign oxide) disappeared.

The relative concentrations of the magnesium oxide surface cleaved in laboratory air are indicated in table II. The relative concentration of adventitious hydrocarbon, which was present on the magnesium oxide surface and was introduced from the laboratory environments, was about 5 at.%. The estimated relative concentrations of magnesium and oxygen were 0.44 and 0.51 before sputtering and 0.47 and 0.53 after sputtering. Again, no carbon was present on the magnesium oxide surface after sputtering.

Hardness measurements were conducted on single-crystal magnesium oxide cleaved in laboratory air and in nitrogen. The Vickers hardnesses on the magnesium oxide {001} surface were 824 and 831, respectively.

Figure 5 presents a typical distribution of dislocation etch pits around an indentation where the specimen was cleaved in air. Because magnesium oxide is highly ionic, slip does not occur on the close-packed {001} cube planes, as already indicated, but instead on the {011} cube diagonals. The actual dislocation etch-pit pattern around the Vickers indentation on the {001} surface contained screw dislocations in the {001} directions and edge dislocations in the {011} directions, as indicated by the pit patterns in figure 5 (ref. 9). The distribution widths of the screw and edge dislocations were 4.9 and 7.7 times larger than the average length of the two diagonals of indentation.

Influence of Mineral Oil and Additives

Indentation experiments were conducted in the various

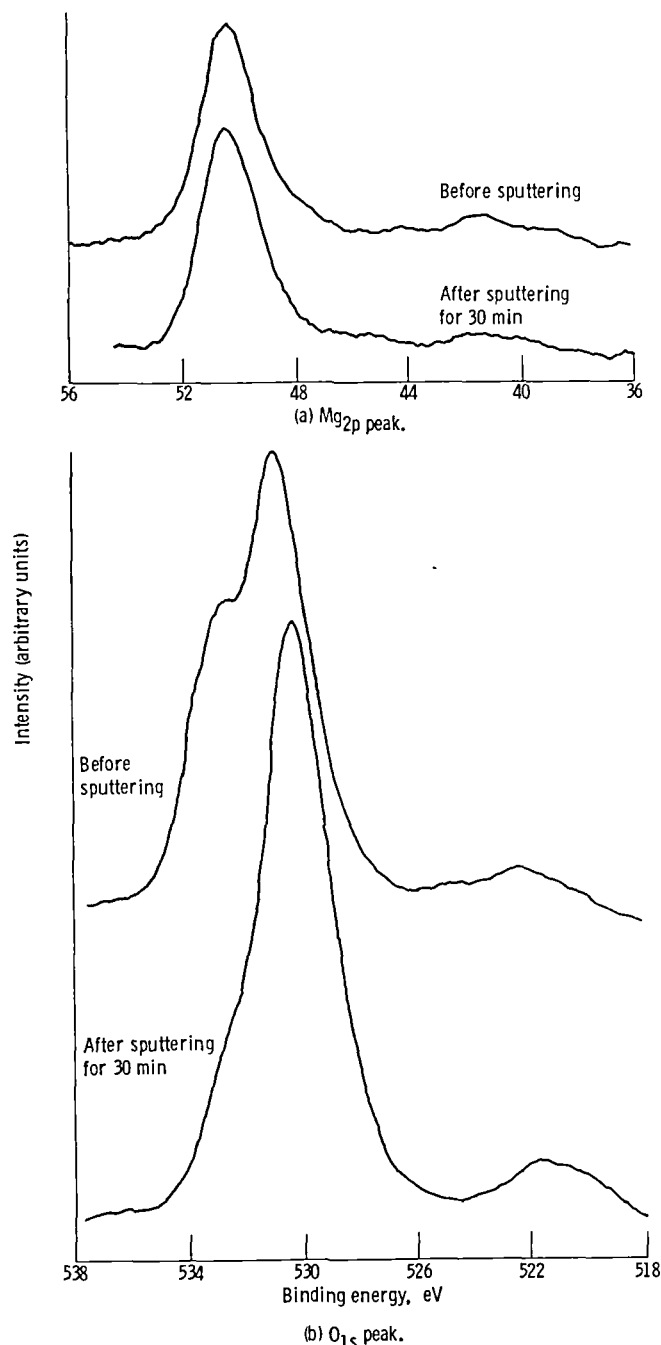


Figure 4. - Representative Mg_{2p} and O_{1s} XPS peaks on magnesium oxide {001} surface cleaved in laboratory air.

environments, that is, in pure mineral oil, and in the mineral oils containing 2 wt % additive with the actual additive element being phosphorus, sulfur, chlorine, or oleic acid. The results presented in figures 6 and 7 indicate that the hardness and distribution widths of the screw and edge dislocations were influenced by the oil and its additives. Each value of microhardness and distribution width of dislocations is the average of 10 measurements. The standard deviations of the

TABLE II. - RELATIVE CONCENTRATION OF CONSTITUENTS ON
MgO SURFICIAL LAYER BEFORE AND AFTER SPUTTERING

Surface treatment before installation in vacuum chamber	Surface treatment in vacuum chamber	Concentration				
		Mg	O	C	Cl	S
Cleaved in air	No treatment	0.44	0.51	0.05	----	----
	Sputtering	0.47	0.53	----	----	----
Cleaved in mineral oil containing chlorine additive	No treatment	0.32	0.30	0.36	0.02	----
	Sputtering	0.51	0.48	Negligible	0.01	----
Cleaved in mineral oil containing sulfur additive	No treatment	0.36	0.31	0.33	----	----
	Sputtering	0.52	0.48	Negligible	----	----

measurements of the microhardness, the distribution width of edge dislocations, and the distribution width of screw dislocations were less than 3.1, 4.1, and 6.5 percent, respectively. Judging from the statistical analysis with the table of Student's t-distribution (ref. 11), the microhardness number and the distribution widths of edge dislocations from the experimental results in mineral oil with sulfur or chlorine additive differed meaningfully from other experimental results obtained with a 95-percent confidence limit. The difference between the distribution widths of screw dislocations for the sulfur-containing additive and those for the chlorine-containing additive was also meaningful according to the same criterion. The variations of the screw and edge dislocation widths clearly correlated. The hardness had a inverse correlation with the distribution widths of the dislocations.

The hardness and dislocation widths of the magnesium oxide {001} surface cleaved in mineral oil without additive were almost the same as those of the surfaces cleaved in air or nitrogen.

The sulfur-containing additive in mineral oil, however, exhibited the highest hardness and the smallest dislocation widths, and the chlorine-containing additive in mineral oil exhibited the lowest hardness and the largest dislocation widths. Hence, to gain an understanding of the surfaces of magnesium oxide cleaved in mineral oil containing sulfur and chlorine additives, XPS analysis was conducted with these magnesium oxide surfaces.

Survey spectra (1000-eV scans) were taken for the surfaces of magnesium oxide cleaved in mineral oil containing the additives. The typical results are shown in figure 8. Figure 8 presents the survey spectra obtained before and after argon ion sputtering for the magnesium oxide surface cleaved in oil with the chlorine additive. The spectrum obtained before sputtering indicates a

carbon contaminant on the magnesium oxide surface as well as magnesium, oxygen, and a very small amount of chlorine. The spectrum taken for the surface after sputtering for 30 min still reveals a carbon contaminant on the magnesium oxide surface in addition to magnesium, oxygen, and chlorine.

The survey spectra were also taken for the surfaces of magnesium oxide cleaved in mineral oil containing a sulfur additive. The spectrum obtained before sputtering indicates a carbon contaminant on the magnesium oxide surface as well as magnesium and oxygen. No sulfur, however, was evident on the surface. The spectrum taken after sputtering for 30 min still reveals a very small carbon contaminant peak. The carbon contaminant was due to the absorption of the mineral oil and the additives as well as hydrocarbon introduced from the laboratory environment.

The results obtained from narrow scans are shown in figures 9 and 10. Chlorine is clearly evident in the spectra both before and after sputtering (fig. 9(a)). The Mg_{2p} and O_{1s} main peaks were seen at 48.8 and 529.5 eV before sputtering and at 50.2 and 530.2 eV after sputtering. The relative concentrations of various constituents present on the magnesium oxide surface cleaved in mineral oil containing the chlorine additive are listed in table II. The relative concentration of hydrocarbon was about 36 at.%, but no carbon was evident after sputtering. The relative concentrations of magnesium and oxygen were 0.32 and 0.30 before sputtering and 0.51 and 0.48 after sputtering. The relative concentration of chlorine was about 2 at.% before sputtering and about 1 at.% even after sputtering. The surface cleaved in mineral oil containing the chlorine additive revealed that magnesium was richer on this surface than it was on the surface cleaved in air.

The results obtained for the magnesium oxide surface cleaved in mineral oil containing the sulfur additive are

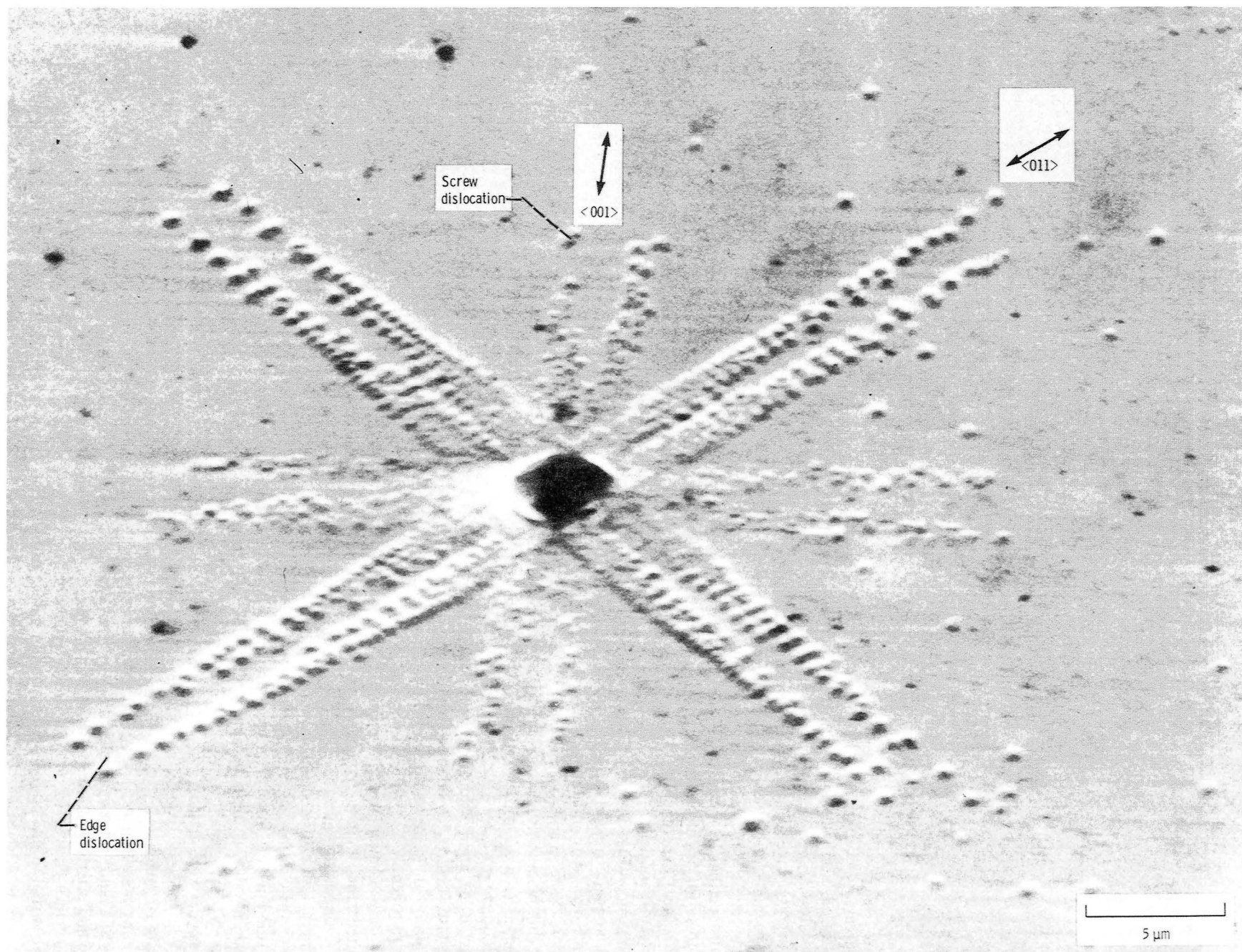


Figure 5. - Distribution of dislocation etch pits around Vickers indentation on magnesium oxide surface. Indenting surface, {001} cleaved in laboratory air; load, 0.1 N; indenter, diamond.

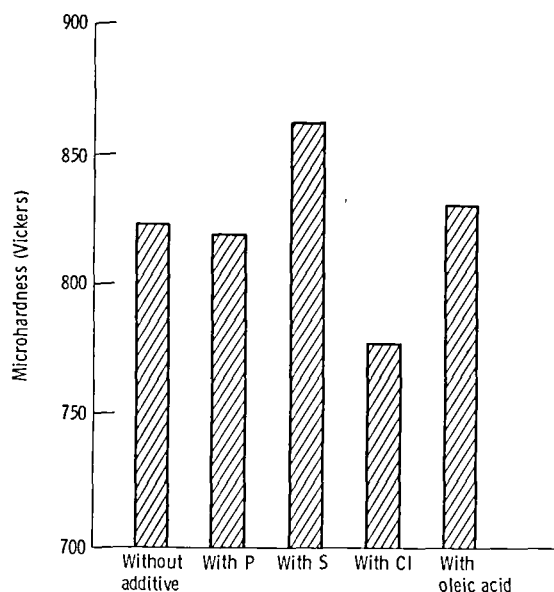


Figure 6. - Microhardness (Vickers) of magnesium oxide {001} surface cleaved (and hardness measurements conducted) in mineral oil with and without various additives. Additives containing phosphorus, sulfur, chlorine, or oleic acid; load, 0.1 N; indenter, diamond; room temperature.

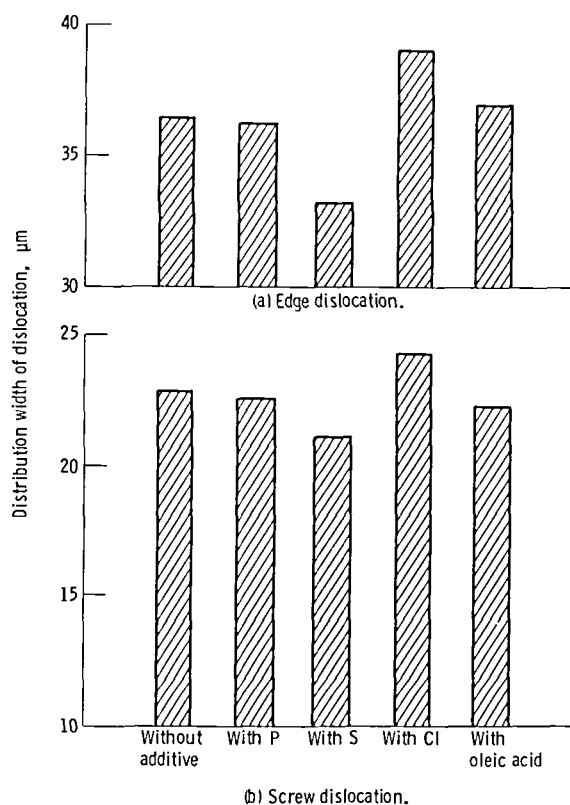


Figure 7. - Distribution widths of edge and screw dislocations around Vickers indentations on cleaved magnesium oxide {001} surface. Indentations were conducted in mineral oil with and without various additives. Additives containing phosphorus, sulfur, chlorine, or oleic acid; load, 0.1 N; indenter, diamond; room temperature.

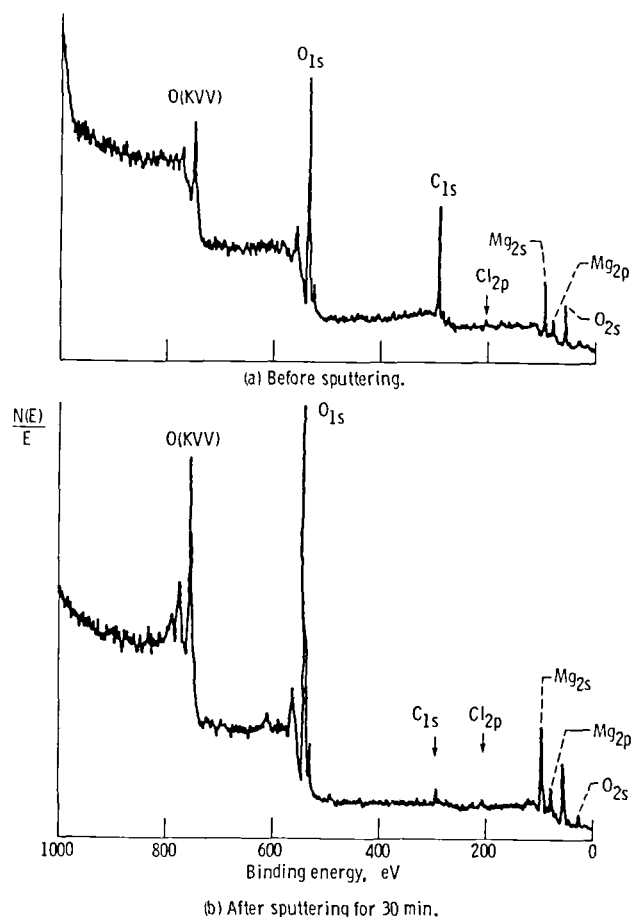


Figure 8. - Survey spectra of magnesium oxide {001} surface cleaved in mineral oil containing chlorine additive and then argon ion sputtered for 30 min.

shown in figure 10. No sulfur is evident in the spectra of the magnesium oxide surface either before or after sputtering. The Mg_{2p} and O_{1s} peaks for magnesium oxide were at 48.9 and 529.4 eV before sputtering and at 50.2 and 530.2 eV after sputtering. The relative concentrations of various constituents present on the magnesium oxide surface cleaved in mineral oil containing the sulfur additive are listed in table II. The relative concentrations of magnesium, oxygen, and carbon were 0.36, 0.31, and 0.33, respectively, before sputtering. Those of magnesium and oxygen were 0.52 and 0.48, respectively, after sputtering.

Thus the hydrocarbon and chlorine films formed as a result of the chlorine-containing additive interacting with the magnesium oxide surface. Chlorine chemically reacted with the magnesium to produce the compound MgCl₂ in the surficial layer. Even after 30 min of sputtering, the compound MgCl₂ was not removed from the surface. The lowest hardness and the largest dislocation widths shown in figures 6 and 7 are believed to be due to the formation of MgCl₂ in the surficial layer. In such a surficial layer, dislocations, which are created by indenting, may move more easily than they do on

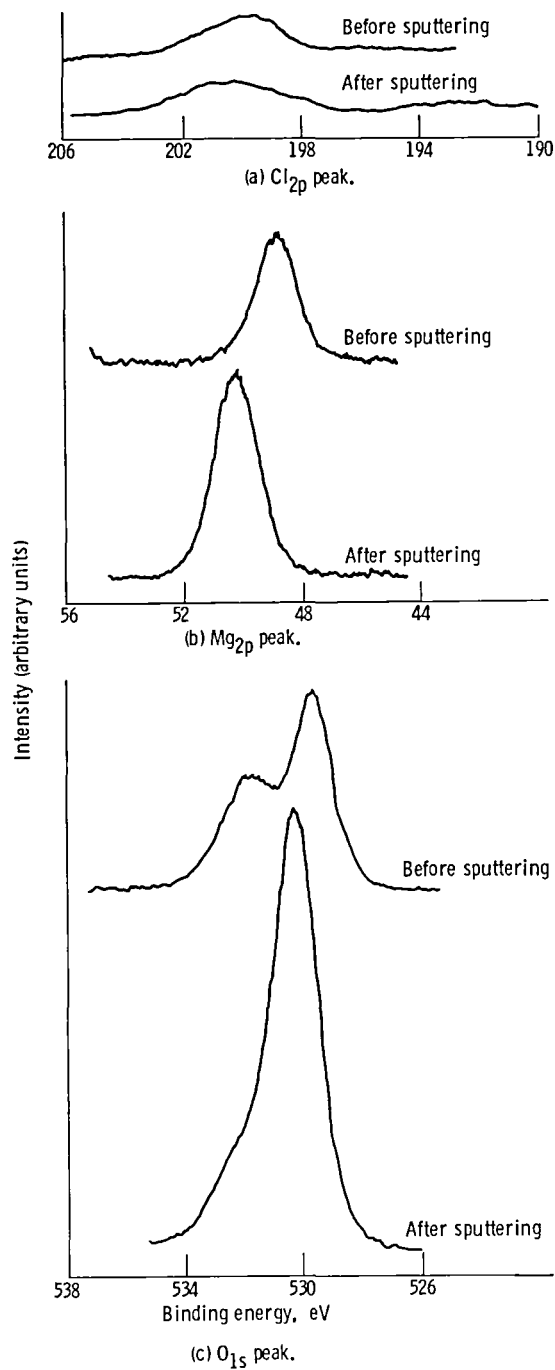


Figure 9. - Representative Cl_{2p}, Mg_{2p}, and O_{1s} peaks on magnesium oxide {001} surface cleaved in mineral oil containing chlorine additive and then argon ion sputtered for 30 min.

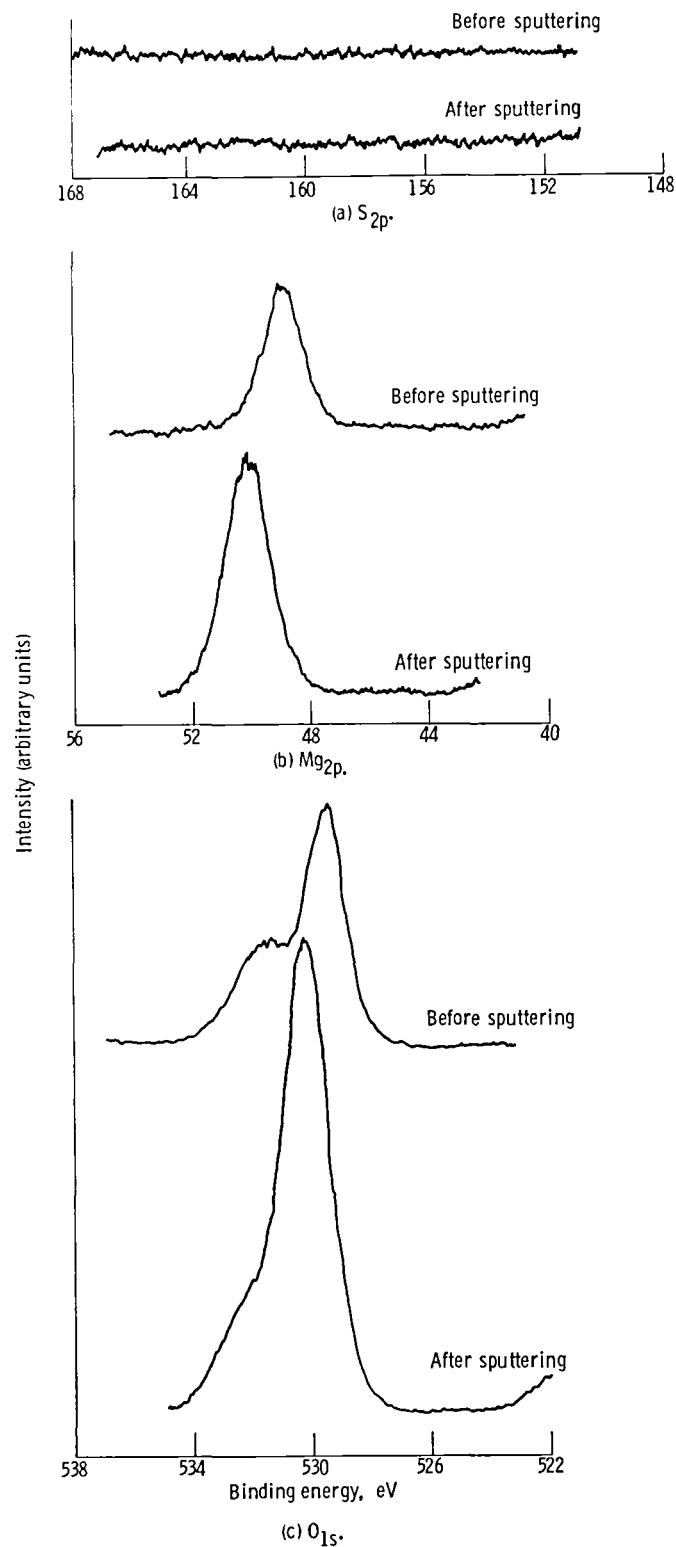


Figure 10. - Representative S_{2p}, Mg_{2p}, and O_{1s} peaks on magnesium oxide {001} surface cleaved in mineral oil containing sulfur additive and then argon ion sputtered for 30 min.

surfaces cleaved in air or in mineral oil containing sulfur, phosphorus, or oleic acid additives.

The hydrocarbon film related to the oil and sulfide formed as a result of a sulfur-containing additive interacting with the magnesium oxide surface. The films may act as a significant energy barrier to the movement of dislocations. In other words, the films may decrease the mobility of dislocations and thus result in the observed increase in hardness. Because dislocations, which are created by indenting, may pile up in the surface layer under the films, the films can raise the stress to move dislocations to the surface and offer resistance to plastic deformation during indenting.

Evidence of the sulfide film was not revealed by XPS analysis. When the specimens were dried and exposed to oxygen in air before being placed in the ultra-high-vacuum system, the sulfur could have been completely displaced from the magnesium oxide surface (ref. 12).

Conclusions

As a result of XPS analyses and hardness measurements conducted with single-crystal magnesium oxide {001} surfaces in various environments, the following conclusions are drawn:

1. Hydrocarbon and chloride (MgCl_2) films are formed on a magnesium oxide surface as a result of a chlorine-containing additive interacting with a cleaved magnesium oxide surface. The indentation on this surface exhibits the lowest hardness and largest dislocation widths around the indentation.

2. Sulfur-containing additives in mineral oil exhibit the highest hardness and smallest dislocation widths around the indentation.

3. The hardness and dislocation widths around an indentation on a magnesium oxide surface cleaved in mineral oil with and without phosphorus or oleic acid are almost the same as those of the surface cleaved in air or nitrogen gas.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, November 20, 1981

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